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(54) Title: ANTIMICROBIAL COMPOSITIONS

(57) Abstract: The invention relates to an antimicrobial cleaning composition which is designed in particular for the hygienic cleaning of hard surfaces. The composition comprises a synergistically active antimicrobial mixture of (i) hydrogen peroxide or a precursor thereof and (ii) picolinic acid; characterised in that the molar ratio of picolinic acid to hydrogen peroxide (or precursor thereof generating the equivalent amount of hydrogen peroxide) ranges from 1:30 to 100:1. The synergistic effects of the picolinic acid with hydrogen peroxide are particularly pronounced against a broad spectrum of microbes.

ANTIMICROBIAL COMPOSITIONS

Technical Field

The present invention relates to an antimicrobial cleaning composition which is designed in particular for the hygienic cleaning of hard surfaces. The composition is based on a synergistically active antimicrobial mixture of hydrogen peroxide and picolinic acid.

Background and Prior Art

Cleaning compositions designed for application to hard surfaces in the household, or in institutional or hospital environments, generally comprise one or more surfactants, and, optionally, one or more antimicrobial actives and/or solvents.

Hard surfaces found in the household, or in institutional or hospital environments, such as kitchen work surfaces and bathrooms, are often contaminated with bacteria and other microorganisms, which present a risk to human health, especially when they are present near food.

The biocidal activity of surfactants is, with a few notable exceptions, low and it is therefore commonplace to add a separate antimicrobial active to compositions.

Hydrogen peroxide is known as an environmentally friendly antimicrobial oxidant, but typically requires long contact times and/or high concentrations in order to be effective.

We have found that picolinic acid acts synergistically in combination with hydrogen peroxide to provide much greater antimicrobial activity than that obtained from the two components individually.

This allows effective hygiene performance to be obtained at much lower levels of hydrogen peroxide than would typically be used in a hygiene product such as an antimicrobial household cleaner.

Advantageously the synergistic effect is not significantly impaired in the presence of protein soil and/or anionic and nonionic surfactants.

Picolinic acid has been described for example in WO9007501 (Solvay Interox)as a stabiliser for percarboxylic acid bleaching compositions. The picolinic acid is added in minor amounts to sequester transition metals which catalyse peroxygen compound decomposition.

WO9700312 and WO9716521 (Procter & Gamble) mention picolinic acid as a suitable ligand for transition metal (e.g. cobalt) complexes which are used in catalytically effective amounts in laundry and machine dishwash formulations to catalyse bleaching by hydrogen peroxide.

US4171313 (Allied Chemical Corporation) discloses the use of picolinic acid and dipicolinic acid as ligands in molybdenum or tungsten complexes which catalyse the oxidation of simple organic molecules by hydrogen peroxide.

US4138488 (Schering Corporation) discloses that diphenylmethyl picolinic acids can kill the Gram positive bacteria implicated in acne in the absence of hydrogen peroxide.

EP0845526 (Eka Chemicals AB) discloses the use of dipicolinic acid or derivatives thereof as complexing agents for obtaining satisfactory storage stability of hydrogen peroxide in an acidic aqueous cleaning composition. It is also stated that the agents are believed to enhance the antimicrobial activity of hydrogen peroxide by chelating magnesium and calcium on the bacterial cell surface. Picolinic acid, 2,6-pyridine dialdehyde and 2,2-dipyridyl amine are mentioned as useful derivatives of dipicolinic acid. The dipicolinic acid or derivative thereof is said to be present at 0.25 to about 5 wt%, preferably from about 0.5 to about 2 wt% based on the content of hydrogen peroxide.

EP066307 (P&G) describes the use of dipicolinic acid for preventing bulging in closed deformable containers containing a peroxygen source such as hydrogen peroxide. The dipicolinic acid is present in a weight ratio of dipicolinic acid to hydrogen peroxide of most preferably 1:10 to 1:800, giving a molar ratio ranging from about 1:4000 to 1:50.

EP 0624640 (P&G) describes dipicolinic acid and picolinic acid as suitable chelants to be used with peroxygen bleach such as hydrogen peroxide. The molar ratio of dipicolinic acid to hydrogen peroxide in the exemplified formulations is typically around 1:100.

EP0634476 (P&G) relates to hard surface cleaners in which dipicolinic acid (or derivatives thereof) is used to build viscosity in aqueous emulsions of nonionc surfactants. Hydrogen peroxide is also present in the examples and the molar ratio of dipicolinic acid to hydrogen peroxide is around 1:200.

US4129517 (Sterling Drug) describes a stable aqueous peroxy-containing concentrate containing percarboxylic acid, hydrogen peroxide and a stabilising agent for the peracid and hydrogen peroxide which is typically a mixture of 2,3-pyridinedicarboxylic acid and 2,6-pyridine dicarboxylic acid. The molar ratio of pyridinedicarboxylic acid to peroxide is typically about 1:400.

WO93/23517(Laporte) describes an antimicrobial formulation comprising phosphoric and/or sulphamic acid, a peroxygen source and a wetting agent. Dipicolinic acid is present in an exemplified composition as a peracid stabiliser and the molar ratio of dipicolinic acid to hydrogen peroxide is 1:88.

US5872092 describes non-aqueous bleach containing liquid laundry detergents which contain perborate or percarbonate as peroxygen precursors. Dipicolinic acid is present in the

exemplified compositions at a level which typically gives a molar ratio of dipicolinic acid to hydrogen peroxide of about 1:100.

None of the above referenced prior art discloses or suggests the combination of ingredients which characterises compositions of the present invention as defined below, and the improved hygienic performance for the cleaning of hard surfaces obtainable thereby.

Summary of the Invention

The present invention provides an antimicrobial cleaning composition suitable for the hygienic cleaning of hard surfaces, which composition comprises a synergistically active antimicrobial mixture of (i) hydrogen peroxide or a precursor thereof and (ii) picolinic acid;
characterised in that the molar ratio of picolinic acid to hydrogen peroxide (or precursor thereof generating the equivalent amount of hydrogen peroxide) ranges from 1:30 to 100:1.

Detailed Description of the Invention

Hydrogen Peroxide

Compositions of the invention contain, as an essential ingredient, hydrogen peroxide or a precursor thereof.

The hydrogen peroxide may be incorporated *per se* into the composition, or may be generated *in situ* by use of a hydrogen peroxide precursor. Examples of suitable hydrogen peroxide precursors are compounds which produce hydrogen peroxide on dissolution in water, (such as sodium perborate monohydrate, sodium perborate tetrahydrate, sodium percarbonate and the urea-hydrogen peroxide addition compound percarbamide), and enzymatic hydrogen peroxide generating systems (such as peroxidases, oxidases and other oxido-reductase enzyme systems, in conjunction with their appropriate substrates).

The amount of hydrogen peroxide in compositions of the invention (or precursor thereof generating the equivalent amount of hydrogen peroxide), suitably ranges from 0.0001% to 5%, more preferably from 0.001 to 1.5%, most preferably from 0.003% to 0.5%, by weight based on total weight of the composition.

Picolinic acid

Compositions of the invention contain, as an essential ingredient, picolinic acid.

The total amount of picolinic acid in compositions of the invention suitably ranges from 0.01% to 10%, more preferably from 0.1% to 5%, most preferably from 0.15% to 2.5%,by weight based on total weight of the composition.

Ingredient Ratios

It is an essential feature of the composition of the invention that the molar ratio of picolinic acid to hydrogen peroxide (or precursor thereof generating the equivalent amount of hydrogen peroxide) ranges from 1:30 to 100:1.

Preferably the molar ratio ranges from 1:30 to 75:1, more preferably from 1:20 to 75:1, most preferably from 1:10 to 50:1.

Product Usage

Compositions of the invention are designed in particular for the hygienic cleaning of hard surfaces. By "hard surfaces" is meant those surfaces which are typically found in the household, or in institutional or hospital environments, and which are prone to microbial contamination. Examples include bathroom fixtures, bathroom appliances (toilets, bidets, shower stalls, bathtubs and bathing appliances), wall and flooring surfaces and those surfaces associated with kitchen environments and other environments associated with food preparation.

The benefits of being able to formulate at reduced levels of antimicrobial active (due to the positive synergistic interaction observed in compositions of the invention) are particularly significant in the context of household cleaning. Such advantages include the reduction or elimination of potential skin or eye irritation associated

with consumer usage of the formulation, and the reduction or elimination of formulation residues which could, for example, contaminate food.

Compositions of the invention may also be useful in laundry or personal care applications where an antimicrobial effect is desirable, such as in deodorant or oral care formulations.

They may also be suitable for food hygiene products such as fruit and vegetable washes, since the component ingredients of compositions of the invention are safe, biodegradable and natural.

Product Form and Packaging

Compositions of the invention may take any form suitable for hygiene products.

If the hydrogen peroxide is to be incorporated *per se* into the composition, then it is preferably in the form an aqueous composition, in which the principal ingredient is water, which is normally present at a level of at least 75%, preferably at least 90%, more preferably at least 95%, by weight based on total weight of the composition. The use of distilled or demineralised water is preferred, but not essential to the invention.

Aqueous compositions of the invention may suitably be supplied as a ready to use product packaged in a

conventional plastics container. Examples of suitable packaging forms include spray dispensers, foam dispensers, toilet cleaner dispensers, and all packaging forms which are adapted to dose the product for neat use. By "neat use" is meant that the product is not diluted by the consumer prior to use. Alternatively the composition may be used by the consumer in diluted form. By "diluted form" is meant that the composition is diluted by the user, typically with water.

Aqueous ready-to-use compositions of the invention will generally have a pH between 2 and 11, more preferably between 3 and 7, most preferably between 3.5 and 6.5.

Alternatively, compositions of the invention may contain a hydrogen peroxide precursor which produces hydrogen peroxide on dissolution in water, in which case a solid product form can be used, such as a tablet.

Other Ingredients

Compositions according to the present invention may further contain any conventional surfactant for emulsification and cleaning purposes. The concentration of this surfactant material is generally in the range from 0 to 50% by weight based on total weight of the composition, but a maximum concentration of 20% by weight based on total weight of the composition is usually preferred. Suitable surfactants may be anionic surfactants, such as alkyl aryl sulfonates, alkyl sulfates and alkyl sulfonates, nonionic surfactants, such as

ethylene oxide and/or propylene oxide condensation products with alcohols or alkylphenol, or mixtures thereof.

The composition according to the invention can contain other optional ingredients which aid in their cleaning performance and maintain the physical and chemical stability of the product.

Hydrotropes are useful optional components. These enable the cloud point of the composition to be raised without requiring the addition of anionic surfactants.

Suitable hydrotropes include alkali metal toluene sulphonates, urea, alkali metal xylene and cumene sulphonates, short chain (preferably C_{1-5}) alcohols, and glycols. Preferred amongst these hydrotropes are the sulphonates, particularly the cumene and toluene sulphonates.

Typical levels of hydrotrope range from 0 to 5% by weight based on total weight of the composition, for the sulphonates. Correspondingly higher levels of urea and alcohols are required. Hydrotropes are not required for dilute products.

Solvents may be present in the compositions of the invention.

Suitable solvents correspond to the general formula R_1-O-$(EO)_m$-$(PO)_n$-R_2, wherein R_1 and R_2 are independently C_{2-6} alkyl or hydrogen, but not both hydrogen, m and n are

independently 0-5, EO represents an ethyleneoxy group and PO represents a propyleneoxy group.

Suitable materials of this type include the C_{1-4} alkyl ethers of alkylene glycols such as ethylene glycol, propylene glycol and oligomers thereof having 2 or 3 repeating units. Examples include monoethers such as ethylene glycol mono-n-butyl ether, ethylene glycol monomethyl ether, ethylene glycol monoethyl ether, ethylene glycol mono-n-hexyl ether, propylene glycol monomethyl ether, propylene glycol monoethyl ether, propylene glycol mono-n-butyl ether, isopropylene glycol monoethyl or monopropyl or monobutyl ether, diethylene glycol monoethyl or monopropyl or monobutyl ether, di- or tripropylene glycol monomethyl ether, di- or tripropylene glycol monoethyl ether, and mixtures thereof.

Typical levels of solvent range from 0 to 2% by weight based on total weight of the composition.

Compositions according to the invention can also contain, in addition to the ingredients already mentioned, other optional ingredients such as pH regulants, sunscreens, foam-control agents, colourants, viscosity modifying agents, freeze-thaw stabilisers, additional hygiene agents, perfumes and opacifiers.

The invention will now be illustrated by the following non-limiting Examples, in which all percentages are by weight based on total weight, unless otherwise indicated.

EXAMPLES

The bactericidal activities of various combinations of hydrogen peroxide and picolinic acid, compared with controls using either ingredient individually, were tested using the European Suspension Test protocol (European standard EN1276). Biocidal test data against two representative bacteria, *E. coli* ATCC 11229 (Gram -negative) and *S. aureus ATCC 6538* (Gram-positive) are shown below in Table 1.

Table 1

Bactericidal activity from combinations of picolinic acid and hydrogen peroxide (30 minutes contact time) in unbuffered systems containing water of standard hardness (pH c.a. 5):

	Mean log reduction in cell numbers (from log-7 cell suspension)			
	E. coli		*S. aureus*	
Formulation	No BSA[3]	0.3% BSA	No BSA	0.3% BSA
0.0034% hydrogen peroxide	0	0	0	0.1
0.123% picolinic acid	0	0.2	0.4	0.2
0.0034% hydrogen peroxide + 0.123% picolinic acid	3.5	3.6	2.9	2.6
0.0034% hydrogen peroxide + 0.123% picolinic acid + 0.01% SDS[1]	5.4	5.2	4.9	2.5
0.01% SDS	0	0	0.5	0.1
0.0034% hydrogen peroxide + 0.01% SDS	0	0	0.5	0.1
0.123% picolinic acid + 0.01% SDS	0.3	0	2.9	0.2
0.0034% hydrogen peroxide + 0.123% picolinic acid + 1% Neodol 91-8[2]	5.4	5.2	2.4	1.7
0.0034% hydrogen peroxide + 1% Neodol 91-8	0	0	0.5	0.3
0.123% picolinic acid + 1% Neodol 91-8	0.9	0.7	0.4	0.3
0.0034% hydrogen peroxide + 0.123% picolinic acid + 0.01% SDS + 1% Neodol 91-8	5.4	5.2	2.3	1.7
0.0034% hydrogen peroxide + 0.01% SDS + 1% Neodol 91-8	0	0	0.5	0.2
0.123% picolinic acid + 0.01% SDS + 1% Neodol 91-8	0.9	0.8	0.4	0.3

(1) SDS - sodium dodecylsulphonate

(2) Neodol 91-8 - alcohol ethoxylate surfactant (C_{9-11}, 8EO) ex. Shell.

(3) BSA - bovine serum albumin (protein soil)

The data shows that bactericidal performance of the picolinic acid/hydrogen peroxide is significantly greater than that observed from the same levels of either picolinic acid or hydrogen peroxide alone (< log 0.5 in the absence of surfactants). In the case of *E. coli*, these log-kill values are comparable to those obtained using 0.34% hydrogen peroxide, i.e. addition of picolinic acid allows a 100-fold reduction in the concentration of hydrogen peroxide required to achieve log 5 reduction in *E. coli* cell numbers.

The data also shows that the synergistic effect between hydrogen peroxide and picolinic acid is largely retained in the presence of protein soil and selected anionic and non-ionic surfactants.

CLAIMS

1. An antimicrobial cleaning composition suitable for the hygienic cleaning of hard surfaces, which composition comprises a synergistically active antimicrobial mixture of (i) hydrogen peroxide or a precursor thereof and (ii) picolinic acid; characterised in that the molar ratio of picolinic acid to hydrogen peroxide (or precursor thereof generating the equivalent amount of hydrogen peroxide) ranges from 1:30 to 100:1.

2. A composition according to claim 1, in which the amount of hydrogen peroxide(or precursor thereof generating the equivalent amount of hydrogen peroxide), ranges from 0.0003% to 5%, more preferably from 0.003% to 1.5%, most preferably from 0.03% to 0.5%, by weight based on total weight of the composition.

3. A composition according to claims 1 or 2, in which the amount of picolinic acid ranges from 0.01% to 10%, more preferably from 0.1% to 5%, most preferably from 0.15% to 2.5%,by weight based on total weight of the composition.

4. A composition according to any of claims 1 to 3, in which the molar ratio of picolinic acid to hydrogen peroxide (or precursor thereof generating the equivalent amount of hydrogen peroxide) ranges from 1:30 to 75:1, more preferably from 1:20 to 75:1, most preferably from 1:10 to 50:1.

5. The use of a composition according to any of claims 1 to 4, for the hygienic cleaning of hard surfaces.

INTERNATIONAL SEARCH REPORT

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A. CLASSIFICATION OF SUBJECT MATTER
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Minimum documentation searched (classification system followed by classification symbols)
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Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)
EPO-Internal, PAJ, WPI Data

C. DOCUMENTS CONSIDERED TO BE RELEVANT

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X	EP 0 906 950 A (PROCTER & GAMBLE) 7 April 1999 (1999-04-07) page 4 -page 5	1-4
A	EP 1 010 750 A (PROCTER & GAMBLE) 21 June 2000 (2000-06-21) page 3; claims	1-5
A	EP 0 624 640 A (PROCTER & GAMBLE) 17 November 1994 (1994-11-17) cited in the application page 6, line 4; claims; examples 2,3; table 1	1-5

☐ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

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